28909 DUP

IGM88TC-401-730-G
CNRC 28909

IMPROVED ULTRASONIC EVALUATION OF GREEN-STATE CERAMICS WITH THE USE OF A SURFACE-BONDED ADHESIVE TAPE

K. Yamanaka*, C.K.Jen, C. Neron and J. F. Bussiere

Industrial Materials Research Institute
National Research Council of Canada
75 Boul. de Mortagne, Boucherville,
Quebec, Canada J4B 6Y4

* Present address: Mechanical Engineering Laboratory,
Namiki 1-2, Tsukuba, Ibaraki, 305 JAPAN

ABSTRACT

An improved method has been developed for the NDE of green-state ceramics where samples are covered with an adhesive tape. The tape serves to protect the sample from contamination by ultrasonic couplant and the adhesive serves to couple ultrasonic waves into the sample. This tape can be removed without any damage to the surface. If the sample is sealed by the tape, water immersion C- scan imaging can be applied. Furthermore, if a water bag is placed onto a taped sample and a transducer is scanned in the water, complete sealing is not necessary, and hence practical industrial testing of large or curved samples may be achieved. This method has been applied to large and thick (70 mm diameter and 19 mm thickness) green-state alumina with 4 % PVA binder. Subsurface features such as an inclusion (2 mm diameter paper sheet at 8 mm depth) and large variations with time of the amplitude and time delay of the back surface echo associated with variations in humidity were recorded by using a 0.5 MHz, 4" focal length transducer. A detailed comparative study between acoustic images before and after sintering was also conducted.

1. INTRODUCTION

Recently, the importance of nondestructive testing of ceramics in the green state before sintering has been recognized because it is economically important to save unnecessary and expensive sintering and machining of defective parts. It is also interesting to investigate the behavior of defects before and after the sintering process. So far, a few methods have been proposed to acheive this goal, however, each of the methods has some drawbacks.

A vacuum coupling method[1,2] was applied to water immersion C scan imaging. This method needs complete sealing of the sample to protect it from water. For this reason, it would not be easy to apply this method to large or complex-shaped samples. Another method proposes to use dry contact by using an elastomer [3,4], although much larger contact pressure is necessary than with ultrasonic couplant. It is difficult to extend this method to C scan imaging or mapping of variations of acoustic property.

As an extension of the above works, a technique has been developed to monitor the velocity change of powder during compaction [5]. Though this method is applicable to process monitoring, it is not applicable to products taken out of the compaction die. It is also difficult to extend this concept to C scan imaging.

In this paper, we propose an alternative method using adhesive tape which enables to use standard ultrasonic couplant for testing green state ceramics. It enables C scan imaging if the

sample is completely covered with a tape. Furthermore, if we use a water bag together with the taping method, no complete sealing is necessary and it is possible to apply to large or curved samples.

2. TECHNIQUES

2.1 Sample Preparation

We prepared a few large and thick (70 mm diameter and 19 mm thick) green alumina discs with 4 % PVA binder (made by Almax Industries Inc., Lindsay, Ont.). They came from a single lot. Sample #3 is a normal sample. Sample #2 has been wetted during a water immersion measurement and sample #4 has a paper sheet inclusion which is about 2 mm diameter in the center of the sample. The density after sintering was 90 % of theoretical density (3.7 g/cm^3).

In order that the time delay of back surface echoes can be converted to velocity, the green state samples were carefully ground into plates with uniform thickness (18.95 $\pm$ 0.05 mm). After sintering they were also ground by a standard procedure into parallel plates with thickness around 15.5 mm.

2.2 Tape Coupling

The procedure of our method is simply to cover the sample with an adhesive tape. The basic idea is to use the tape to protect the sample from contamination by ultrasonic couplant, whereas the adhesive allows good coupling of ultrasonic waves into the sample as shown in Fig. 1. We tried two types of tapes; polypropylene and polyester. In both cases the thickness of the tape itself is

28 μm and the thickness of the adhesive is 22 μm. The tape can be removed without any damage to the sample. Pictures of green state samples with and without tape are shown in Fig. 2.

2.3 Contact Transducer

Standard contact transducers with gel couplant between the transducer and the sample covered with a tape were used to measure acoustic wave velocities in the central area of the sample. The measured longitudinal wave velocity of sample #1 with a 25.4 mm diameter transducer at 0.5 MHz was 1580 m/s, whereas shear wave velocity was around 1080 m/s. Then the Poisson's ratio is estimated to be 0.06 which is quite low and typical for brittle materials.

2.4 Laser Ultrasound

One of our approaches for inspecting green state ceramics is based on laser generated ultrasound which has the advantage of being non-contact [6]. Ultrasound is generated by a high energy pulse laser and detected by the laser interferometer. First, we examined the feasibility of using laser generated ultrasound by evaluating the strength of signals transmitted through the thick samples in a set-up similar to Fig. 1. The generating laser (Laser Science PRF 150G CO_2 laser, with a pulse energy of 0.28 J, pulse duration of 100 ns, repetition rate of 6 pps and beam diameter of 3mm) was radiated onto the top surface of the sample and the transmitted acoustic waves were detected by the transducer under the sample (Panametrics V101 transducer with 0.5 MHz center frequency and 25.4 mm diameter) and recorded by a Tektronix 7854 oscilloscope. We used a gel couplant (Echo

Laboratories, Sonotrace 20) between the transducer and the sample. The signal was very small and only the first transit pulse was visible as shown in Fig. 3(a) even after 32 times averaging. However, when the top surface was covered with the same tape as the bottom surface the amplitude of the signal increased by 10 dB (Fig.3(b)). Thus, tape enhances the efficiency of the laser generation of ultrasound.

In order to have a completely noncontact method, a laser interferometer employing a 1 Watt Argon laser [6] was then used to replace the conventional piezoelectric transducers shown in Fig.1 as the ultrasonic receiver. Due to the high acoustic attenuation in ceramics, the sensitivity of our current interferometer [6] could not detect any signal even when the taped sample was coated with a layer of retroreflective coating. It is concluded that although laser ultrasound has the advantage being noncontact, a more powerful laser would be required for the interferometer receiver to increase sensitivity for the above decribed green state ceramics samples. Since this was not available at the time, an alternative method described below was explored in more detail.

2.5 Water bag C-scan method

In general, techniques which allow ultrasonic C-scan imaging are preferred to evaluate green state ceramics. Using the above decribed tape, specimen can be carefully sealed and standard water immersion C-scanning techniques applied. We have also developed another approach using a water bag constructed with a

polymer sheet and a metal frame. The sheet material should be as soft as possible to adapt to the sample shape. A 25 μm thick polyethylene sheet turned out to be satisfactory for this purpose. Samples are pressed lightly to the bottom of the water bag and commercial ultrasonic couplant was applied between the taped sample and the water bag. Finally, excess couplant was squeezed out from the interface by using a plate to achieve uniform ultrasonic coupling. Figure 4 shows a photograph of the experimental setup with the water bag apparatus.

3. COMPARISON OF ACOUSTIC PROPERTIES BEFORE AND AFTER SINTERING

3.1 Experimental Procedures

It is of great interest to study the acoustic properties before and after sintering and in the hope that some correlation can be found. A focussed transducer (Panametrics A398; center frequency 0.5 MHz, diameter 1.5" and focal length 4") was used for the green state sample together with the water bag. Figure 5 is an example of the pulse echo waveforms. Between the front surface and the backsurface echo, an echo from a inclusion is visible. By using the cross correlation method [7], time delay of the backsurface echo is evaluated and velocity of longitudinal waves are calculated. Also, by setting a time gate after the surface echo with a suitable threshold, time delay and amplitude of the first echo subsequent to the surface echo are recorded to make subsurface images of the sample. Figures 6 to 8 are the delay time mapping of the first echo following the surface echo, which turns out to be the back surface echo except in defect areas which produce echoes larger than the threshold. The brightness of

each dot corresponds to the amplitude of the echo. The vertical axis corresponds to time delay and the positive direction corresponds to smaller time delay.

After sintering, a plane wave transducer (KB- Aerothech, ALPHA 5 MHz, 0.25 " diameter) was used for the water immersion measurement. A 25 MHz transducer was also tested, but the amplitude of echoes from major defects turned out to be smaller than when the 5 MHz transducer was used. A few degrees of tilt of the transducer with respect to the sample surface was also important to enhance the amplitude of defect echoes.

After the nondestructive inspection, samples were carefully cut by a low speed diamond saw to reveal the ultrasonically detected defects.

3.2 Normal Sample (#3)

The intensity of the back surface echo for the Sample #3 in green state is very uniform as shown in Fig. 6(a). This means that both the coupling of acoustic waves and the attenuation of this sample are uniform. There is a slight curvature of the image showing that the region closer to the edge has a larger time delay than the center region. The variation of longitudinal velocity measured through one diameter of the sample by the correlation method was around $\pm$ 0.9 % as shown in Table I.

After sintering, the image made by the back surface echo seems quite uniform and no defect was detected in the region far from the edge as shown in Fig. 6(b). The variation of velocity for longitudinal waves was also small and only $\pm$ 0.13 % within the

sample (8752 ± 12 m/s) , which is significantly smaller than that in the green state.

3.3 Sample With A Paper Sheet Inclusion (#4)

In order to evaluate the resolution of our technique, sample #4 has a paper inclusion at the center of the sample as described in Section 2.1. Using the method described in Section 3.1 this inclusion caused an earlier reflection from the back surface of the sample, thus a hole in the center of the back surface echo mapping as shown in Fig.7(a). The full width at half maximum of the amplitude mapping of this echo was 6 mm. Since the spatial resolution of the present focussed beam is similar to this value, the size of the inclusion is estimated to be much smaller than 6 mm, which agrees with its actual size of 2 mm.

In contrast to the sample #3 shown in Fig.6(a), the back surface echo in Fig.7(a) near the center of the sample came later than the echo near the edge as indicated by the curvature of the back surface echo mapping. The magnitude of this variation was around ± 2.6 %. A question arises whether this variation in time delay is due to a change in the velocity of the material or due to the scattering of acoustic waves by the inclusion. It might be possible that the embedding of the inclusion during the compaction of the sample caused stress distribution which affected the acoustic property (density or elastic modulus) variation along the radius of the sample.

After sintering, the echo from the inclusion was also clearly detected as shown in Fig. 7 (b). It was found by destructive

inspection that this echo came from a planar defect (4 mm diameter and 1 mm thick) which has an origin at the paper sheet inclusion. The variation in velocity was decreased to ± 0.08 % after sintering.

3.4 Sample That Had Been Wet (#2)

Sample #2 originally had a similar acoustic image to sample #3. However, during a trial of water immersion test by sealing the sample with a tape, water penetrated through the discontinuity of the tape. Then, a smooth gradient of the amplitude and time delay of the back surface echo appeared as shown in Fig. 8(a). Then we took off the tape and dried the sample at ambient temperature for six days. When we observed the same sample with the water bag method, the image was quite different as shown in Fig.8(b). The back surface echo was observed only in a limited area and the time delay was very non-uniform and kept changing with time. The final image before sintering is shown in Fig. 8(c).

Fig.9(a) gives a 2-D representation of Fig.8(c) and a few typtical A-scans near the center of Fig.9(a). Each scan is seperated by a distance of 10 mm. It can be seen that the amplitude of the back surface echo was much smaller in the lower portion of the sample than in the upper portion. Though this feature is very different from other two samples, the optical appearance of the sample surface was quite similar to other samples. The question about this sample is whether this variation is due to a small amount of humidity inside the sample or due to

changed material properties, such as density variation, or to defect formation as a result of wetting.

After sintering, it was found that the back surface echo became significantly uniform as shown in Fig.8(d), though there were many distinct defect echoes such as shown in Fig.9(b) which gives the 2-D images of Fig.8(d) and a few A-scans at locations indicated by the defects A, B, C and a spot without any defect. The sources of these echoes were identified by cutting the sintered sample where strong echoes were observed. Then it was found that there are large (~1 mm) voids as shown in SEM picture of Fig.10.

4. DISCUSSIONS

4.1 Insertion Loss

To evaluate the insertion loss caused by the tape, we have prepared a sintered alumina sample partly covered with the tape and the gel plus polyethylene sheet. The back surface echo obtained with pulse echo measurement by a KB-Aerotech ALPHA 2.25 MHz and 5 MHz immersion transducer was then recorded and the FFT spectrum was analyzed. We define an insertion loss as

$$L = -20 \log(V_a / V_c) \qquad (1)$$

where V_a and V_c are the magnitude of the echo spectrum through the region with and without the tape, respectively. As shown in Fig. 11, the loss is proportional to frequency, which is typical of visco elastic materials. At 0.5 MHz where most measurements for our thick (19 mm) samples were conducted, it is estimated to be 0.7 dB by extrapolating the data in Fig. 11.

It is of interest to note that on the region where the sample is covered only with gel and polyethylene sheet (Region B), the insertion loss was negligible up to 10 MHz. This is probably due to that a decrease of the impedance mismatch between the alumina sample and the water which compensates for the increased attenuation within the sheet. This implies that the present water bag method is very likely applicable at high acoustic frequencies to evaluate other porous materials which do not require the tape, provided that the material is not degraded by gel ultrasonic couplant.

4.2 Comparison Between Water Bag - Tape, X-ray And Laser Ultrasound Methods

To compare the advantage of the present method and other possible methods for green state ceramics, we tried a micro-focus X ray inspection for sample #4, with 50 μm focus on tungsten target, operated at 40 kV and 0.25 mA. Within the sensitivity of 1 % which is acheived by using an emulsion film without digital image processing, this paper inclusion was not detected. To be as sensitive as the ultrasonic method, improved image processing in X-ray radiography would be necessary.

Since sensitivity is the primary concern for those highly attenuating samples, the laser ultrasound methods are not as convenient as the piezoelectric method. However, if we need acoustic images of samples during heating, the non-contacting all laser methods [6] could be better than piezoelectric methods.

4.3 Correlation Of Acoustic Properties Between Green State And Sintered Samples

As shown by the error bars in Fig. 12, the velocity within a sample shows considerable variations in the green state. In particular, there were distinct features, for instance, the velocity was smaller in the center than close to the edge for sample #4 as shown in Fig.7(a), whereas it was opposite in sample #3 as shown in Fig.6(a). After sintering the velocity became very uniform thoughout the samples as shown in Fig.s 7(b) and 6(b) and shown by the error bars in Fig.12.

The attenuation was measured by a FFT spectrum method [8] using a plane wave 25 MHz transducer. It was not significantly different among samples as shown in Table I, considering the uncertainty involved in attenuation measurements of low loss materials. Through inspection of SEM images like Fig. 10, the grain structure of sintered samples was found to be similar for all samples, supporting the attenuation measurement. There are many small voids with 10 to 80 um diameter.

The appearance of acoustic images for the previously wetted sample changed drastically before and after sintering. The effect of wetting the sample seems to be limited only to the odd acoustic property of the green state samples. However, since the large (~1 mm) defects only appeared in the previouly wetted sample, it might be possible that these were formed as a result of local dissolution of the organic binder during the wetting. Further study is necesary to obtain final conclusion on this problem.

5. CONCLUSIONS

An improved method has been developed for the NDE of green state ceramics with the use of a surface-bonded adhesive tape. It is quite useful if a water bag is used together to make C scan images. Insertion loss of the tape is proportional to the frequency and is as low as 0.7 dB at 0.5 MHz, which is adequate for thick (19 mm) green alumina samples. As an application of this method, thick alumina samples with different process history were investigated before and after sintering. This method was also shown to be potentially applicable to evaluate elastic property of materials where ultrasonic couplant should be avoided because of possible degradation by the couplant or the variation of acoustic property due to penetration of liquid.

ACKNOWLEDGEMENT

We appreciate the collaboration of Almax Inc., Lindsay, Ontario and Dr. M. Sayer of Queen's University for providing green state samples and sintering them. We acknowledge Drs. J-P Monchalin and J.D. Aussel of IMRI for the use of laser ultrasound facilities in this work. We also thank Jeffrey Yu for his collaboration in computer programming and Jin Pan for taking SEM pictures. One of the authors (K.Y) was supported by the Summit Postdoctoral Fellowship of National Research Council of Canada.

REFERENCES

1. W.A. Ellingson and R.A. Roberts, Nondestructive testing for structural ceramics, "AR&TD Fossil Energy Materials Program Quarterly Progress Report, 7-9/84, ORNL/FMP-84/4 (1984).

2. R.A. Roberts, Ultrasonic NDE of green-state ceramics by focused through transmission, Rev. Prog. Q-NDE, 6B, 1443-1452(1987).

3. D.S. Kupperman and H.B. Karplus, Ultrasonic wave propagation characteristics of green ceramics, am. Ceram. Soc. Bull. 63(12), 1505-1509(1984).

4. M.P Jones, G.V. Blessing and C.R. Robbins, Mater. Eval. 44, 859-862(1986).

5. M.P. Jones and G.V. Blessing, Real-time ultrasonic evaluation of green state ceramic powders during compaction, Nondestructive Testing Communications, 2(5),155-168(1986).

6. J.-P. Monchalin, J.-D. Aussel, P. Bouchard and R. Heon, "Laser-ultrasonics for industrial applications", Review of Progress in Quantitative NDE, Vol.7B, (D.O. Thompson and D.E. Chimenti eds), pp.1607-1614, Plenum 1988.

7. C.J. Chung, C.K. Jen, J.D. Aussel, J.F. Bussiere and J. C.H. Yu, 'An ultrasonic velocity and attenuation measurement system utilizing a PC controller', to be published.

8. E.P. Papadakis, K.A. Fowler and L.C. Lynnworth, 'Ultrasonic attenuation by spectrum analysis of pulses in buffer rods: method and diffraction corrections', J. Acoust. Soc. Am., Vol.53, pp.1336-1343, 1973.

Figure captions (Green state ceramics)

Fig.1 Principle of tape coupling method

Fig. 2 Picture of samples
(a) after grinding (without a tape)
(b) covered with a tape.

Fig. 3 Transmitted signal of laser-generated ultrasound showing the improvement of the generation efficiency by the tape of 10 dB
Fig. 4 Water bag specially designed for the C-scan imaging.
Water bag method
(a) Schematic illustration
(b) Photograph

Fig. 5 Pulse echo waveforms in focussed transducer measurement using the water bag.

Fig. 6 Acoustic images of normal sample .
(a) Before sintering
(b) After sintering

Fig. 7 Acoustic images of sample with a paper inclusion.
(a) Before sintering
(b) After sintering

Fig. 8 Acoustic images of wet sample.
(a) In wet condition,
(b) After drying for 6 days at ambient temperature,
(c) Before sintering,
(d) After sintering.

Fig. 9 Detailed comparison of previously wetted sample between before and after sintering. (a) Highly attenuated back surface echo in green state and (b) very large defect echoes of sintered sample.

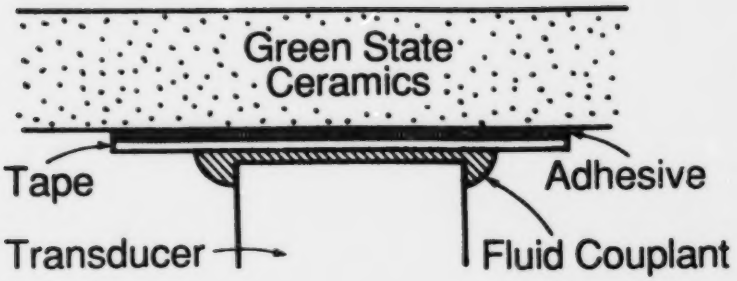
Fig. 10 SEM image of one of the large voids in previously wetted sample.

Fig. 11 Insertion loss introduced by the adhesive tape on a green alumina sample.

Fig. 12 Velocity of longitudinal waves of different samples before ,●, and after 'o' sintering. Circles are averaged values whereas bars represent maximum and minimum within each sample.

TABLE I VELOCITY AND ATTENUATION

Sample Description	#3 normal	#4 with a paper sheet inclusion	#2 had been wetted in green state
Green State Velocity m/s	1644±16 (0.9%)	1529±39 (2.6%)	1760±150 (8.5%)
Sintered Velocity m/s	8752±12 (0.13%)	8743±7 (0.08%)	8762±16 (0.2%)
Sintered Attenuation at 25 MHz dB/cm	2.34	1.90	1.81



Adhesive Tape Method

Fig.1 Principle of tape coupling method

Green Alumina Specimen

(a)

Specimens Covered with Adhesive Tapes

(b)

Fig. 2 Picture of samples
(a) after grinding (without a tape)
(b) covered with a tape.

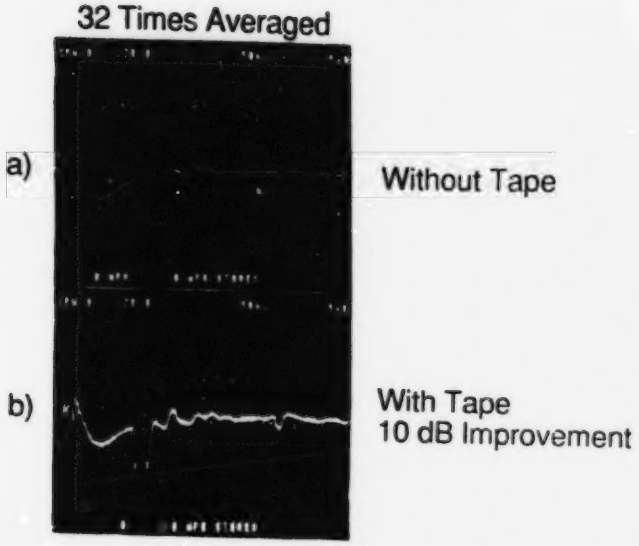
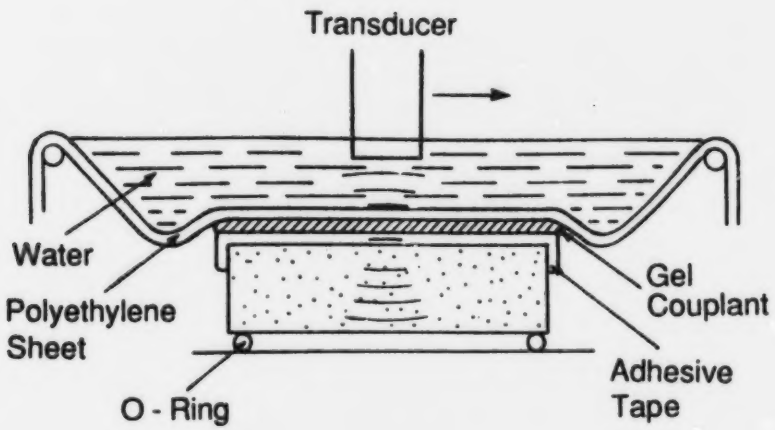
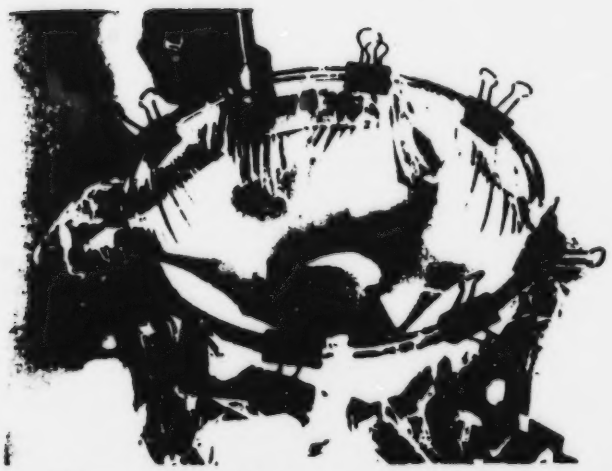


Fig. 3 Transmitted signal of laser-generated ultrasound showing the improvement of the generation efficiency by the tape of 10 dB



(a)



Water Bag Placed onto a Specimen

(b)

Fig. 4 Water bag specially designed for the C-scan imaging.
Water bag method
(a) Schematic illustration
(b) Photograph

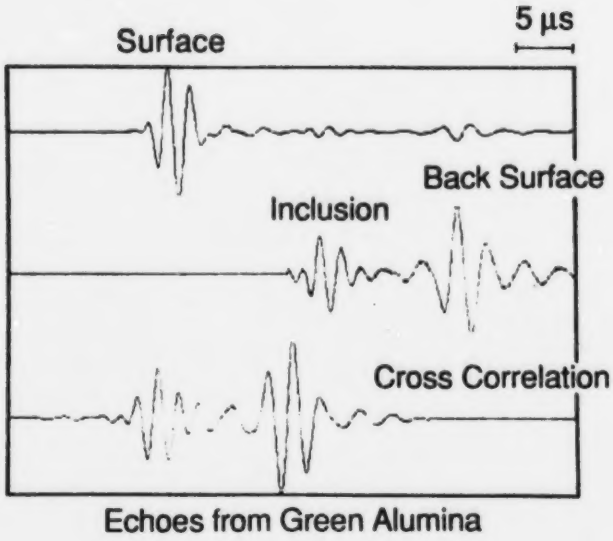
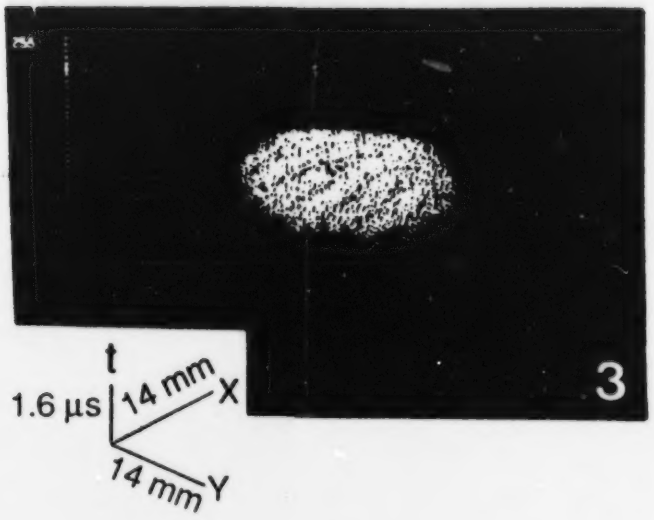
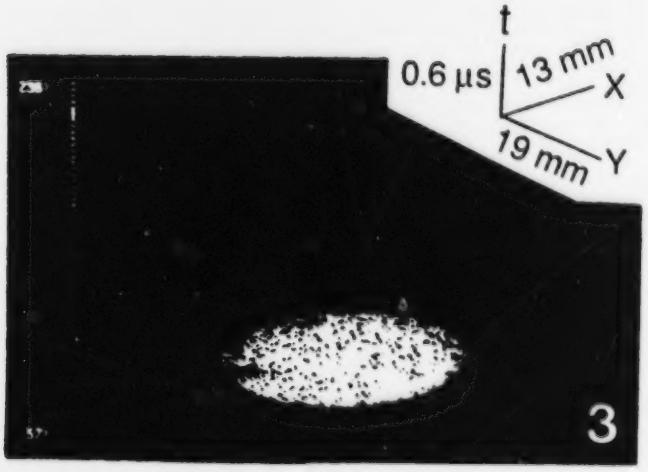


Fig. 5 Pulse echo waveforms in focussed transducer measurement using the water bag.

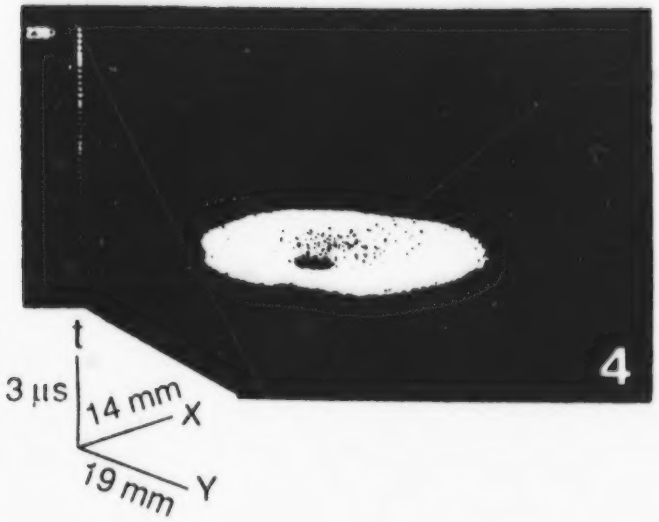


(a)

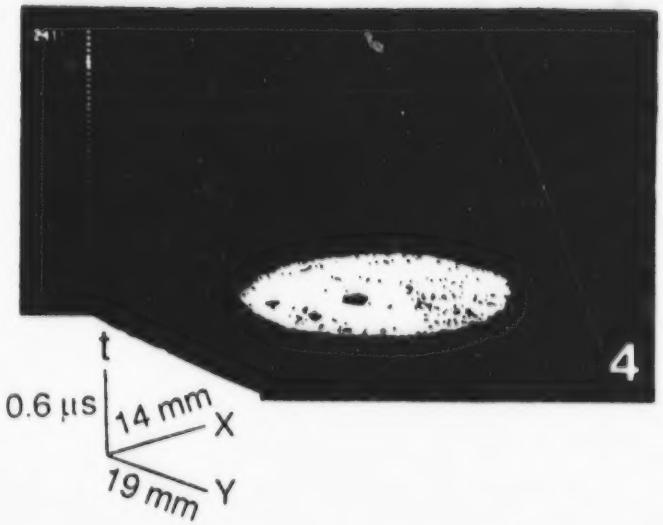


(b)

Fig. 6 Acoustic images of normal sample
(a) Before sintering
(b) After sintering

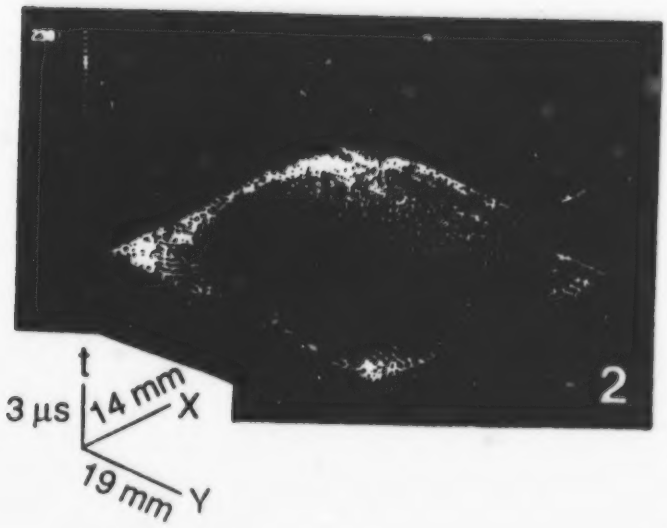


(a)

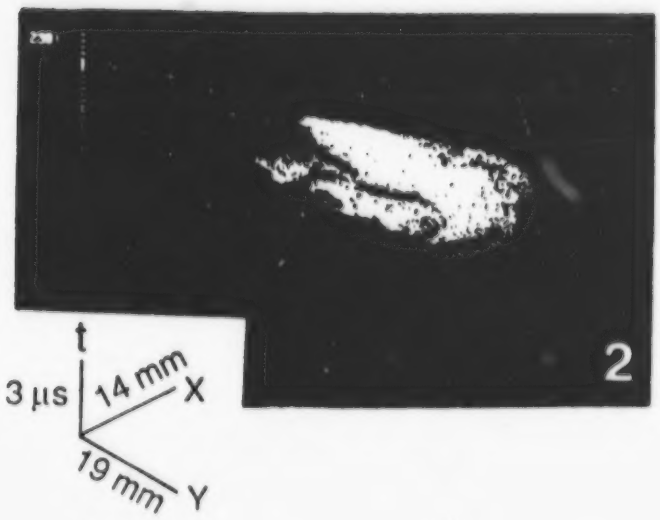


(b)

Fig. 7 Acoustic images of sample with a paper inclusion.
(a) Before sintering
(b) After sintering

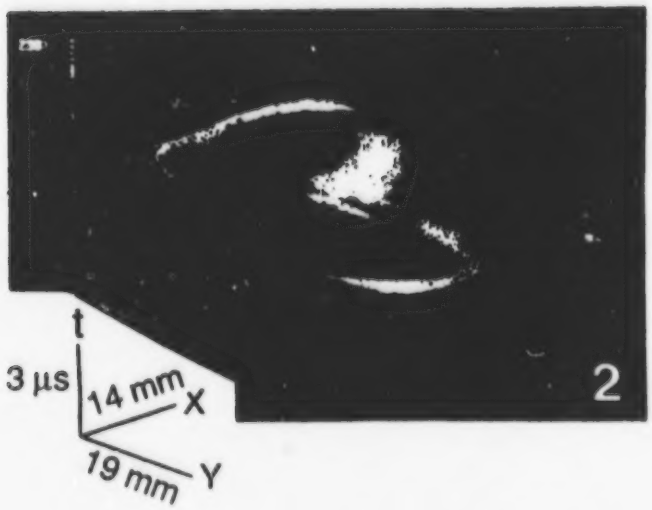


(a)

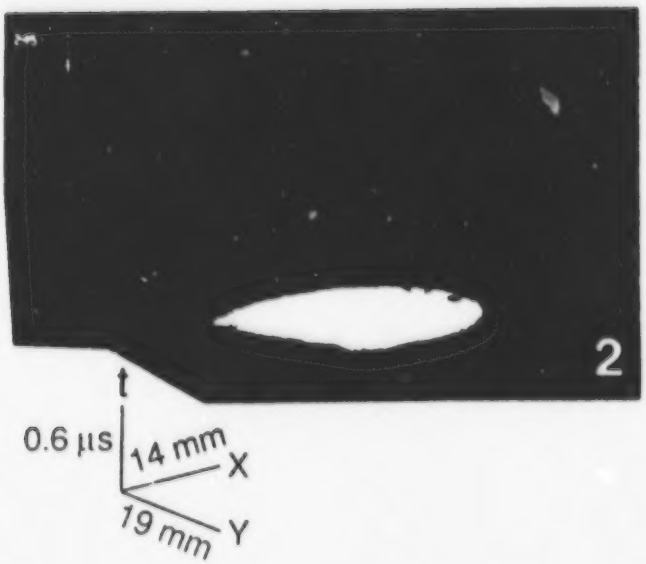


(b)

Fig. 8 Acoustic images of wet sample.
(a) In wet condition,
(b) After drying for 6 days at ambient temperature,
(c) Before sintering,
(d) After sintering.



(c)



(d)

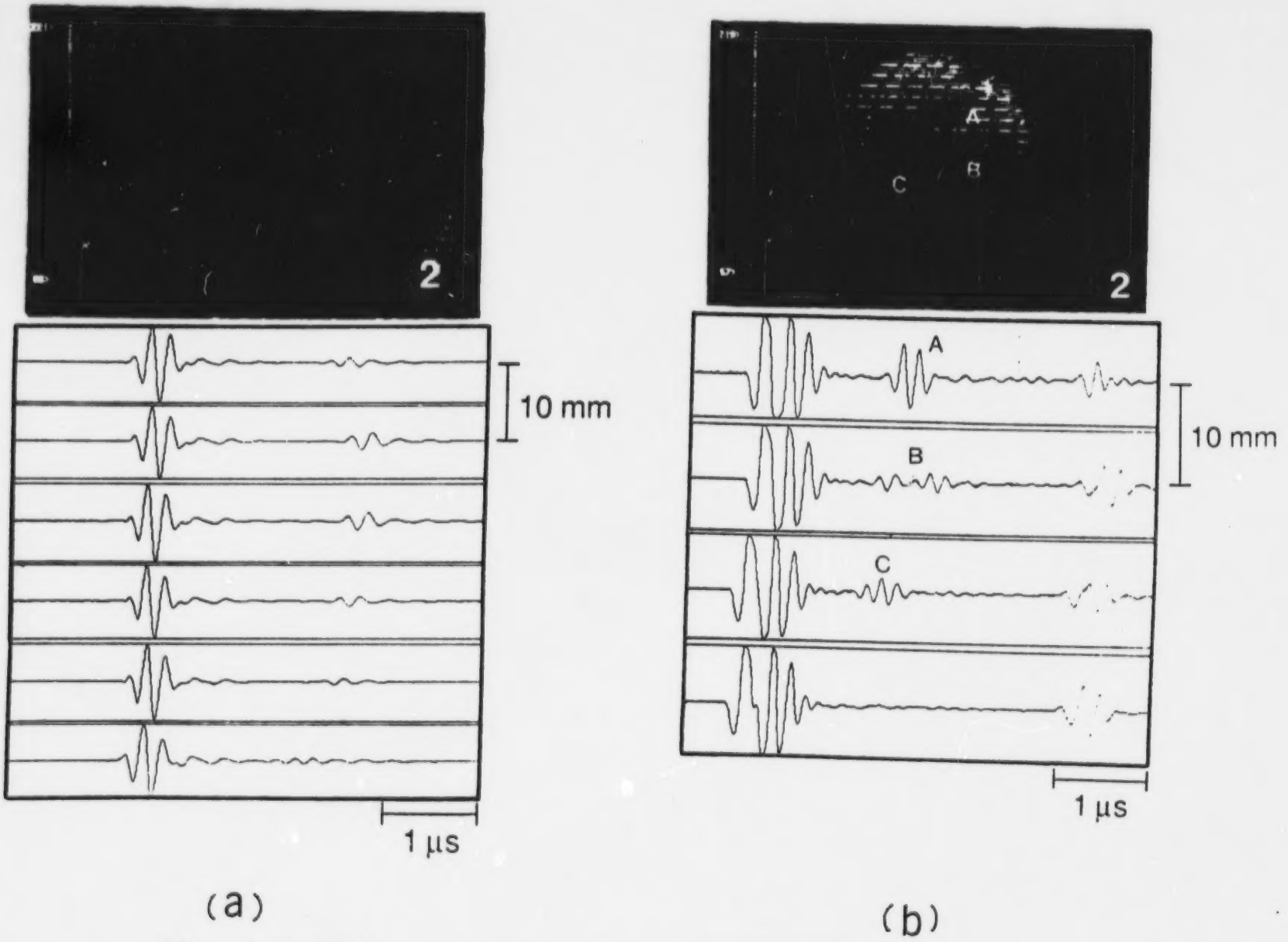


Fig. 9 Detailed comparison of previously wetted sample between before and after sintering. (a) Highly attenuated back surface echo in green state and (b) very large defect echoes of sintered sample.

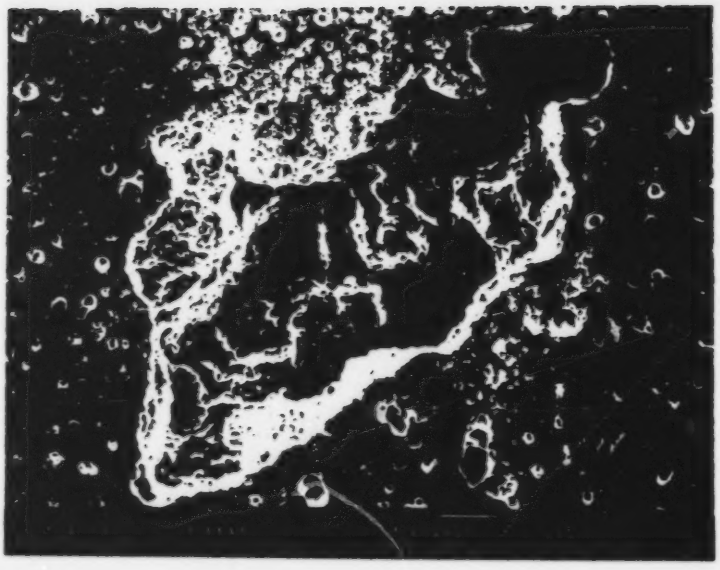


Fig. 10 SEM image of one of the large voids in previously wetted sample.

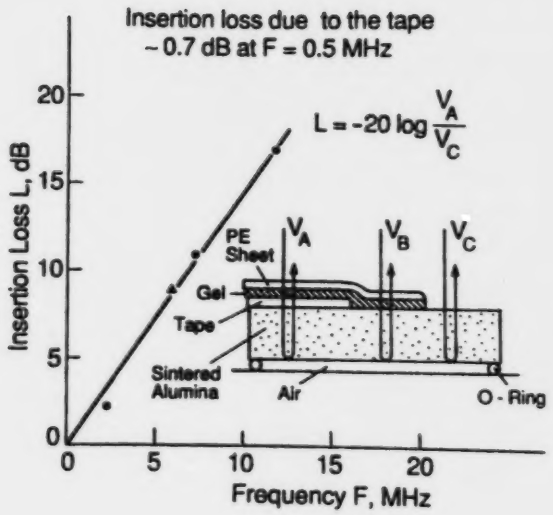


Fig. 11 Insertion loss introduced by the adhesive tape on a green alumina sample.

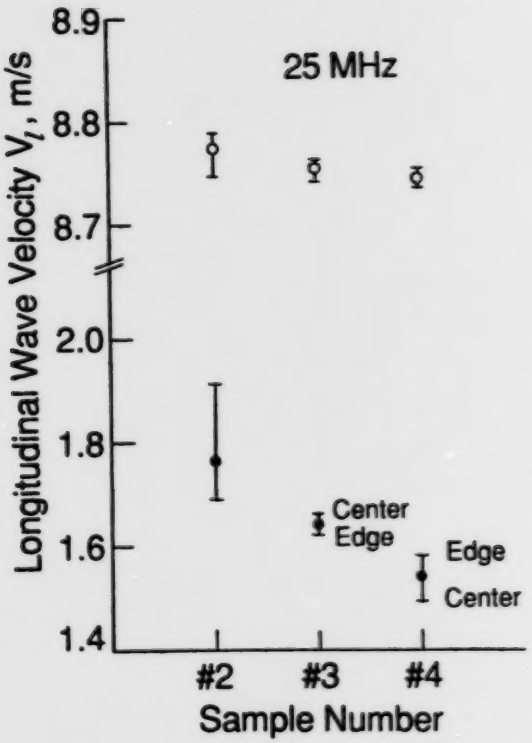


Fig. 12 Velocity of longitudinal waves of different samples before ,●, and after 'o' sintering. Circles are averaged values whereas bars represent maximum and minimum within each sample.